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LIMITED OIL GASIFICATION EXPERIMENT

INTERNATIONAL MATERIALS CORP.

PREPARED FOR
ENVIRONMENTAL PROTECTION AGENCY

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LIMITED OIL GASIFICATION EXPERIMENT

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LIMITED OIL GASIFICATION EXPERIMENT

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16. Abstracts: The report documents the results of a limited oil gasification program to experimentally investigate the feasibility of using International Materials Corporation's Segas system for processing No. 2 and 4 fuel oils and No. 6 high-sulfur residual oil. Existing hardware, originally designed to operate on gasoline for automotive applications, was modified for these tests. The process consists of a high-temperature petrochemical reaction in which a liquid hydrocarbon is thermally decomposed and then reformed to a synthesis gas in the presence of high-temperature steam. Although all tests were not completed by the end of the contract, sufficient progress was made and sufficient results obtained to indicate that the process is capable of stable sustained operation.

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*Segas process
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Object

This Limited Oil Gasification Program is an experimental investigation of the International Materials' SEGAS™ system processing No. 2 and No. 4 fuel oils and No. 6 high sulfur residual fuel oil. The experiment was designed to evaluate the feasibility of gasifying a range of fuel oils and to evaluate the product gas for use as a non-pollutant fuel gas.

Summary

Tests were run to evaluate the capability of the SEGAS oil gasification process on No. 2 and No. 4 fuel oil and on No. 6 high sulfur residual oil. Existing hardware, which was designed to fit within the envelope constraints of an automotive application and to operate on unleaded automotive gasoline, was used as the test reformer. Hardware modifications were kept to the minimum consistent with the program's objectives.

The reformers consist of three finned tubes connected in series and mounted in the hot gas outlet of a burner. A boiler-superheater combination, mounted over the reformer and located in the same hot gas stream, supplied the steam to the reformer for the petrochemical process within the reformer.

The calibration and endurance programs were completed with qualified success on No. 2 fuel and were essentially completed successfully on No. 4 fuel oil. In the former case, the endurance test produced a carbon buildup that is thought to have been caused by operating off the test point because of inadequate control of the process rather than because the test point had not been properly established in the calibration run.

The calibration program on No. 6 high sulfur residual oil was not completed by the end of the contract's period of performance. A problem had been experienced in injecting the oil into the reformer in such a way as to prevent its coking at the entrance. Several combinations of fuel nozzles, steam injection techniques, reformer packing, and steam and reformer temperature variations were investigated; and significant progress was made as evidenced by decreased rate of coke buildup. However, satisfactory operating conditions for the endurance test were not established.

Samples of the product gas were taken at specific times throughout the endurance test programs. Some of these samples were analyzed

by a gas chromatograph at International Materials and the remainder were sent to an outside laboratory for an independent analysis.

Results

1. The SEGAS^{TM} process did not adequately reform No. 2 fuel oil on this program. This was evidenced by an erratic reformer pressure drop during the endurance test, condensable deposits in the output plumbing from the reformer and a carbon deficient analysis of the process based on a chemical analysis of the fuel and a chromatographic analysis of the output gas.

2. The test on No. 4 fuel oil was essentially successful in that the six hour endurance run produced no buildup in reformer pressure drop and subsequent inspection showed no evidence of carbon accumulation in the reformer or any significant amount of condensables in the output plumbing. However, the mass balance performed on the basis of the chemical analysis of the fuel and the chromatographic analysis of the output gas shows a small carbon deficiency.

3. The calibration test on No. 6 high sulfur residual was not completed in that conditions were not achieved that produced a non-deficient process analysis or that showed no accumulation of carbon in the reformer or of condensables in the output plumbing. However, excellent progress was made toward achieving stable operation of the system in that runs of over one-half hour had been made before the reformer pressure drop became excessive.

Conclusions

1. The increasing reformer pressure drop during the endurance run on No. 2 fuel oil suggests carbon accumulation in the reformer. This is because the rate of decomposition of the hydrocarbon fuel is greater than the rate of gasifying. These rates can be brought into balance by changing the rates of heat transfer into the process gas and/or by lowering the pressure at which the reaction takes place. The condensable hydrocarbons in the output plumbing and the failure to achieve a mass balance on the process show that the process had not been carried to completion. This can be corrected by increasing the time at which the reacting constituents are at temperature which can be accomplished by increasing the length of the reaction tubes and/or modifying process pressure. Therefore, it is felt that with additional effort the prospects of meeting the program objectives

with No. 2 fuel oil are very good.

2. The lack of carbon buildup in the reformer on the No. 4 fuel oil endurance test indicates that the process was being conducted in a state of equilibrium. The slight mass unbalance and the small amount of oily deposit in the product gas plumbing suggests that the process was not quite going to completion, in that there were condensable hydrocarbons in the product gas. This can be corrected by slightly increasing the time at which the process constituents are at the reacting temperature, which requires that the length of the reformer be increased and/or that process pressure be increased. Thus, only a slight increase in reformer length may be required to meet the program objectives with No. 4 fuel oil.

3. The increasing reformer pressure drop experienced on the No. 6 high sulfur residual oil calibration test indicates that the run was not made at equilibrium conditions. For successful operation these conditions will have to be established and the determination of reformer length to carry the process to completion will have to be made.

Recommendations

1. The decomposing and reforming reaction characteristics of hydrocarbon fuels suggest that the program objectives can be met by operating the reformer at different temperature and pressure conditions than were used and by increasing the length of the reformer. It is recommended that the investigation be continued in this direction and, when satisfactory operating conditions are established, the endurance test should be rerun.

2. The program on No. 4 fuel oil was essentially completed successfully. The only objective not completely met was that the process was not quite carried to completion as was shown by a mass balance analysis and which was verified by a light oily deposit in the product gas plumbing. This suggests that the length of the reformer and/or process pressure should be increased. It is recommended that the calibration test be run using a longer reformer and, when satisfactory operating conditions are established, the endurance run should be repeated.

3. The progress experienced in establishing acceptable operating conditions with No. 6 high sulfur residual oil suggests that the prospects are good for meeting the program objectives. Therefore, it is suggested that the calibration program be continued and, when satisfactory operating conditions are established, a six hour endurance run should be made.

Discussion

1. SEGAS^{TM} Process

The SEGAS process is a high temperature petrochemical reaction in which a liquid hydrocarbon is thermally decomposed and, in the presence of high temperature steam, is reformed into a synthesis gas product, the composition of which depends somewhat on the conditions at which the process is operated. The criteria of acceptable performance is that the reformer should run in stable equilibrium indefinitely and that the process should be carried to completion, i.e. no condensable hydrocarbons in the output product.

The cracking process is pressure and temperature dependent, the rate increasing with an increase in either of these parameters. This process takes place throughout the reformer as the fuel decomposes progressively from higher hydrocarbon compounds to carbon with the addition of heat.

The reforming process is temperature and pressure dependent, the rate increasing rapidly with increasing temperature and, consistent with the LeChatelier-Braun principle, decreasing somewhat with increasing pressure. This process takes place in the presence of an oxidizer, high temperature steam, which is injected into the reformer with the fuel. Thus, with the rate of cracking increasing with temperature and pressure and the rate of reforming increasing with temperature and decreasing with pressure, the regime of satisfactory reformer operation is bounded by a complex temperature-pressure relationship.

In addition to the chemical reaction considerations, there is the thermal transfer consideration. The SEGAS process may be described as one that operates at high temperature and requires a substantial heat input. Because of the high temperature, most of the heat transfer is by radiation. Radiant heat transfer is time related and so the heat transfer to the reacting constituents is increased with the length of residence time in the radiating environment. Since residence time increases with process pressure, radiant heat transfer also increases with process pressure. This, then, makes it desirable to operate the process at the maximum pressure consistent with the tolerance of the petrochemical reaction. Because process completion is residence time dependent, the use of existing small reformers was damaging to this program in that these reformers were not large enough to provide the residence time needed to completely gasify the No. 2 and No. 4 fuel oils and the No. 6 high sulfur resid oil run on this program.

2. Test Procedure

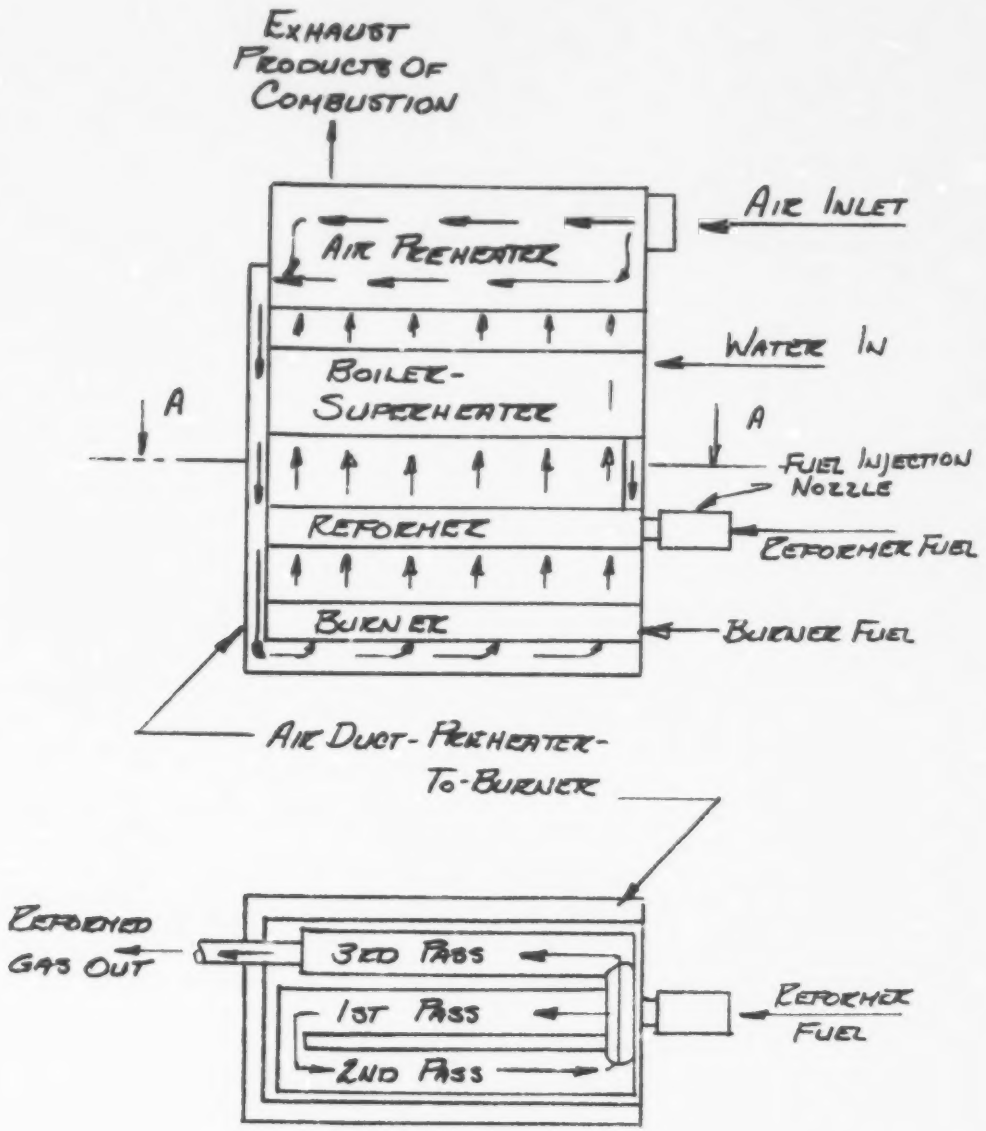
The reformer was set up for the No. 2 fuel oil test on No. 1 test facility at International Materials and was pressure checked preparatory to operation. The test stand instrumentation consisted of: pressure sensors, temperature sensors, flow sensors, pressure regulators, and flow regulators -- all of which were checked out and calibrated prior to the test. With the test facility in order, the first phase of the test program, the calibration run, was started. This was an exploratory program in which acceptable conditions were established for the endurance run with the reformer operating at an acceptable steam-fuel ratio of 4 to 6:1.

With the operating conditions established in the calibration run, the endurance test was started. The reformer was brought to the established conditions and data points were recorded at about 15 minute intervals. The data read were: time of reading, fuel and air flow to the main burner, fuel flow to the reheat burner, oil flow and water flow to the reformer, temperature of the steam into the reformer, of the reformer tube, of the gas out of the reformer, and pressures of the steam at the mixing chamber, of the fuel at the nozzle and of the product gas at the reformer outlet.

The same calibration and operating procedures as used for No. 2 fuel oil were used for No. 4 fuel oil. The calibration test was in process on No. 6 high sulfur residual oil when the contract's period of performance ended.

3. Test Hardware

The reformers that were used on this program were designed to provide the fuel demands and to fit within the envelope constraints of an automotive application and to operate on unleaded automotive gasoline. A sketch of the reformer is shown in Figure 1. The feedstock is sprayed through a fuel injection nozzle into the mixing chamber at the input end of the reformer where it mixes with high temperature steam prior to passing through the reformer and being decomposed and reformed. The reformer consists of three high temperature alloy tubes connected in series by short crossover pipes. Thus, as the reforming process proceeds, the fuel and steam pass through all three tubes. The reforming process is endothermic and so heat must be supplied continuously. This is provided by an external burner in which gas and air are mixed and burned and which is located under the reformer tubes. The products of combustion pass around the reformer tubes which are finned to encourage convective heat transfer in addition to the radiant heat transfer that would normally occur. After the products of combustion have heated the reformer tubes, additional burner gas is supplied to a reheat burner to raise the temperature to that required by the boiler superheater which produces steam for the reformer.



SECTION A-A

- REFORMER -

FUNCTIONAL DIAGRAM

FIG 1

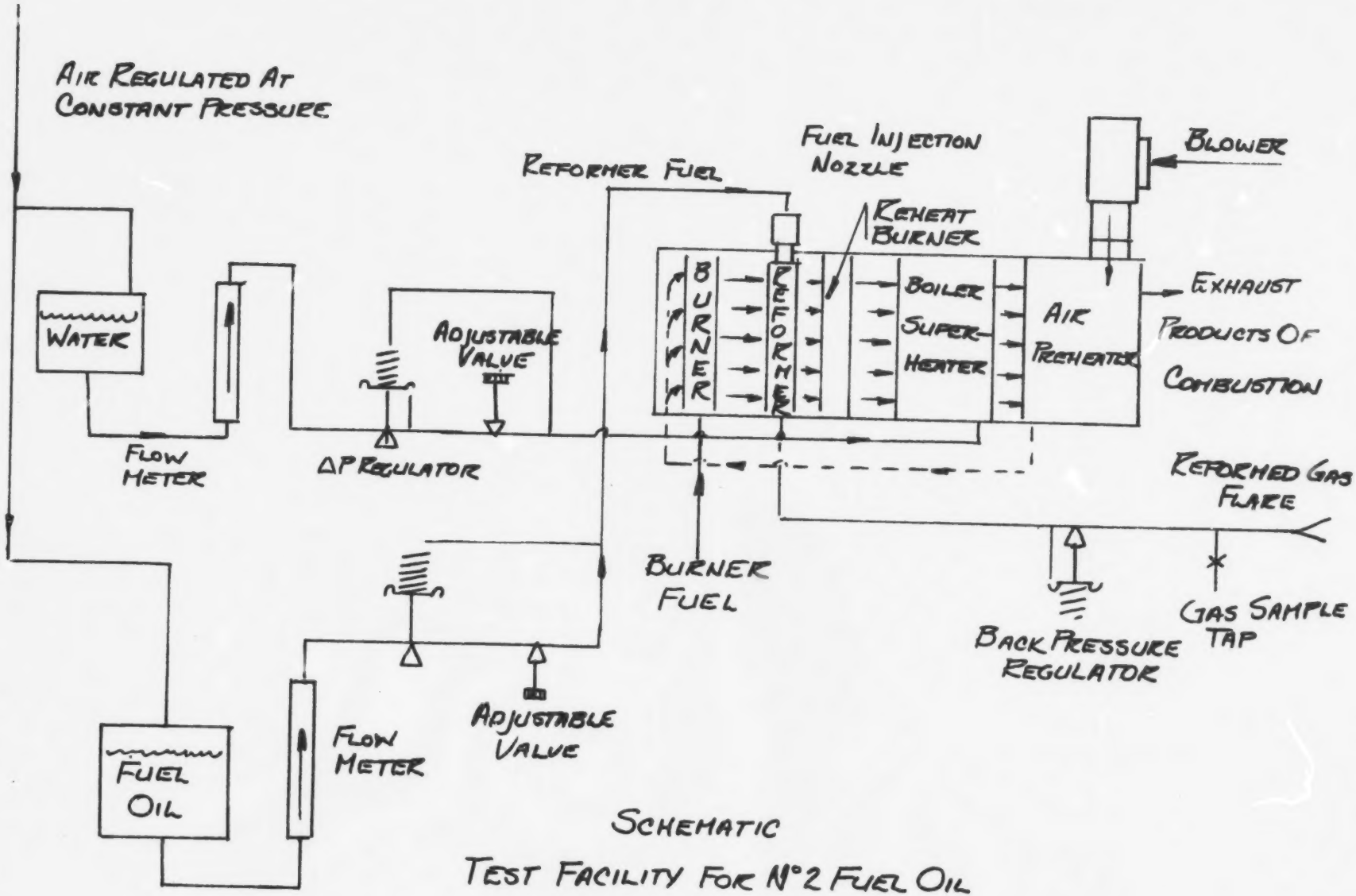
The products of combustion then pass through and release energy to the steam superheater. From the superheater the products of combustion pass through the air preheater, releasing heat energy to and raising the temperature of the air being supplied to the burner. The products of combustion are then exhausted to the atmosphere. The calibration procedure was to bring the reformer to operating temperature with steam flowing and then to turn the fuel oil on. The oil flow, steam flow and temperature and output gas temperature were then adjusted to operate the reformer at the maximum temperature where it would be in stable equilibrium. Complete readings and gas samples were taken. This procedure was run on No. 2 and No. 4 fuel oils and was being run on No. 6 high sulfur residual oil when the contract's period of performance ended.

The reformer tubes are filled with non-catalytic packing material which receives radiant energy from the tube walls and provides heat transfer surface for the reaction. This material also promotes turbulent flow and aids in mixing the reacting constituents. After the gas is processed through the reformer, it passes out through a condenser where the residual water is separated out. The reformed or product gas then passes through a back pressure regulating valve which serves to control the pressure at which the process is operating. The product gas then passes through a flare tube where it is burned or from which test samples are drawn.

4. Test Facility

It was initially intended that the Limited Oil Gasification Experiment would be run on a time sharing basis with other programs in an existing facility. With this arrangement it soon became apparent that schedule conflicts would cause excessive delays in the program. Therefore, a separate test facility was built on International Materials funds for the Limited Oil Gasification Experiment and has been used exclusively for this work. In addition, a portion of this program was run in the facility originally intended. Schematic drawings of the two test facilities are shown in Figures No. 2 and 3.

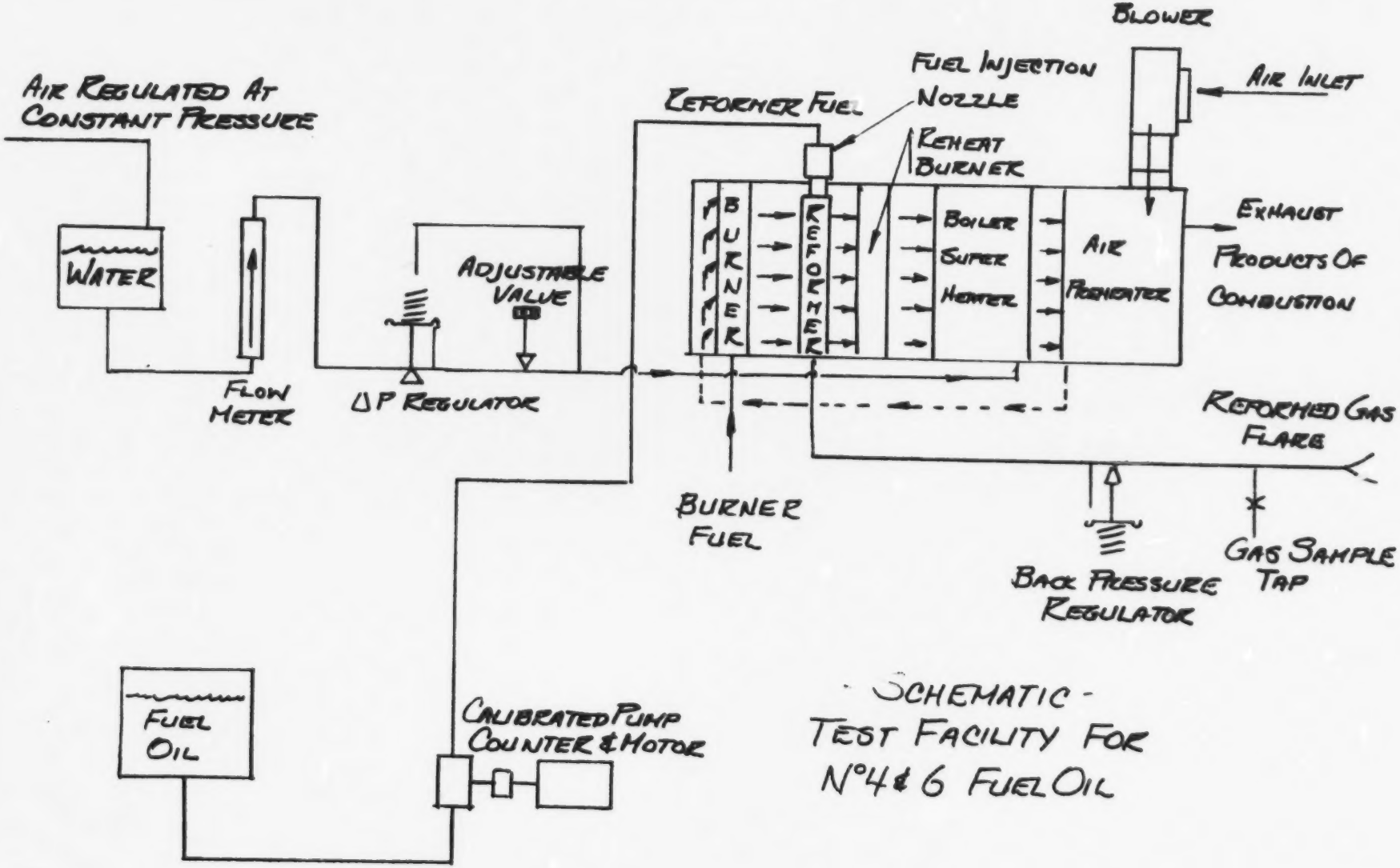
These facilities are similar in that they contain systems for controlling and measuring: the flow of oil to the reformer, of water to the steam generator, of fuel and air to the reformer burner, and of measuring temperatures and pressures: of the steam into the reformer, of the gas out of the reformer and of the temperature of the reformer tube. The primary difference between the facilities is in the manner in which fuel flow is measured. In the original test facility the fuel flow was measured by means of rotameter flow sensors which indicate flow visually by the position of a



SCHEMATIC

TEST FACILITY FOR N°2 FUEL OIL

FIG 2



- SCHEMATIC -
TEST FACILITY FOR
N°4 & 6 FUEL OIL

FIG 3

ball in the flowing fluid stream. The fuel supply was from a tank charged with regulated air pressure to supply the flow producing force. On leaving the tank, the fuel passes through the rotameter flow sensor and then to the flow control system which consists of a micrometer adjustable valve across which the pressure drop is controlled by a throttling regulator valve. Thus, the flow through the system is a function of the opening of the adjustable valve and is unaffected by variations in upstream or downstream pressures in the system.

Because the new test facility was intended to be used for the Limited Oil Gasification Experiment in which opaque No. 4 fuel oil and No. 6 high sulfur residual oil would be run, a visual flow sensing system such as a rotameter was not acceptable. It was decided to use, as the flow sensor, a calibrated positive displacement pump driven by a variable speed electric motor and equipped with a speed counter. The pump was calibrated over the range of flows and backpressures that were anticipated for the test program by flowing the output into a graduated flask for a specific period of time. The runs were repeated and it was found that consistent calibrations were obtained and that, within the limits of observation accuracy and over the range that was to be used on the test program, the system was insensitive to backpressure. The calibration curve is shown as Figure 4.

The gas supply to the reformer main and reheat burners was from a regulated constant pressure source and was measured with a rotameter. The calibration curves are shown as Figures 5 and 6. The flow was controlled by means of a throttling micrometer valve. The flow of air to the burner was supplied by a turbo blower and was determined by measuring the pressure drop across an orifice of known size. The calibration curve is shown as Figure 7. Control was by adjusting the position of a waste gate located between the blower and the sensing orifice. Thus, if the pressure drop across the orifice was too large, indicating excessive flow to the burner, the waste gate opening could be increased to bypass a portion of the blower output to the atmosphere. In the converse case, the waste gate opening would be decreased to reduce the wastage and increase the flow to the burner.

The temperature of the steam leaving the steam generator and entering the reformer was measured by means of sheathed chromel-alumel thermocouples projecting, at right angles, into the flow stream paths. The reformer tube temperature was measured using an intrinsic platinum-platinum 10% rhodium thermocouple and the product gas temperature was measured by a sheathed thermocouple inserted into the product gas outlet of the reformer.

The reformer output flow was passed through a cooling coil to

K•Σ 7 X 10 INCHES KEUFFEL & ESSER CO. MADE IN U.S.A.

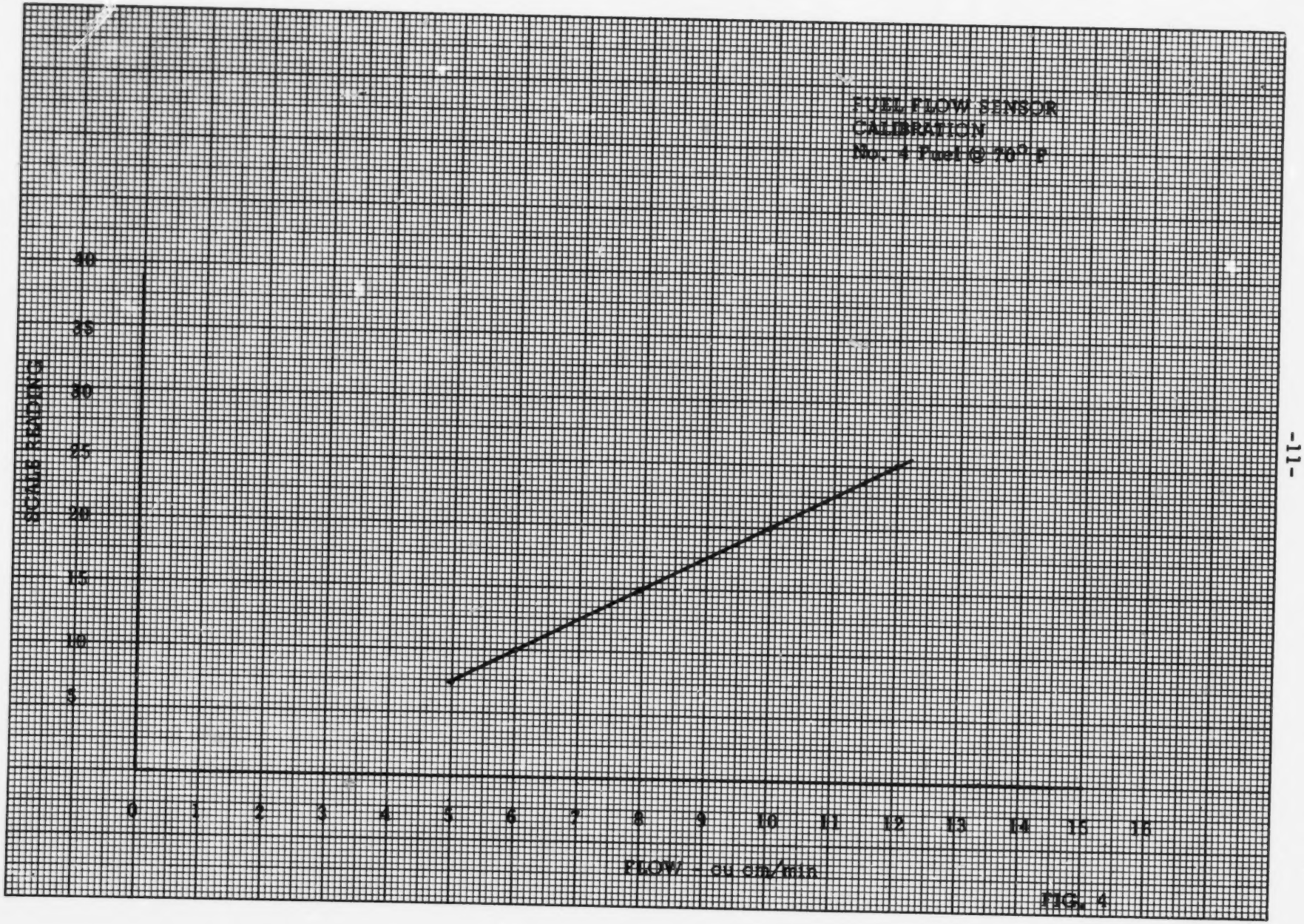


FIG. 4

KEUFFEL & ESSER CO.
MADE IN U.S.A.

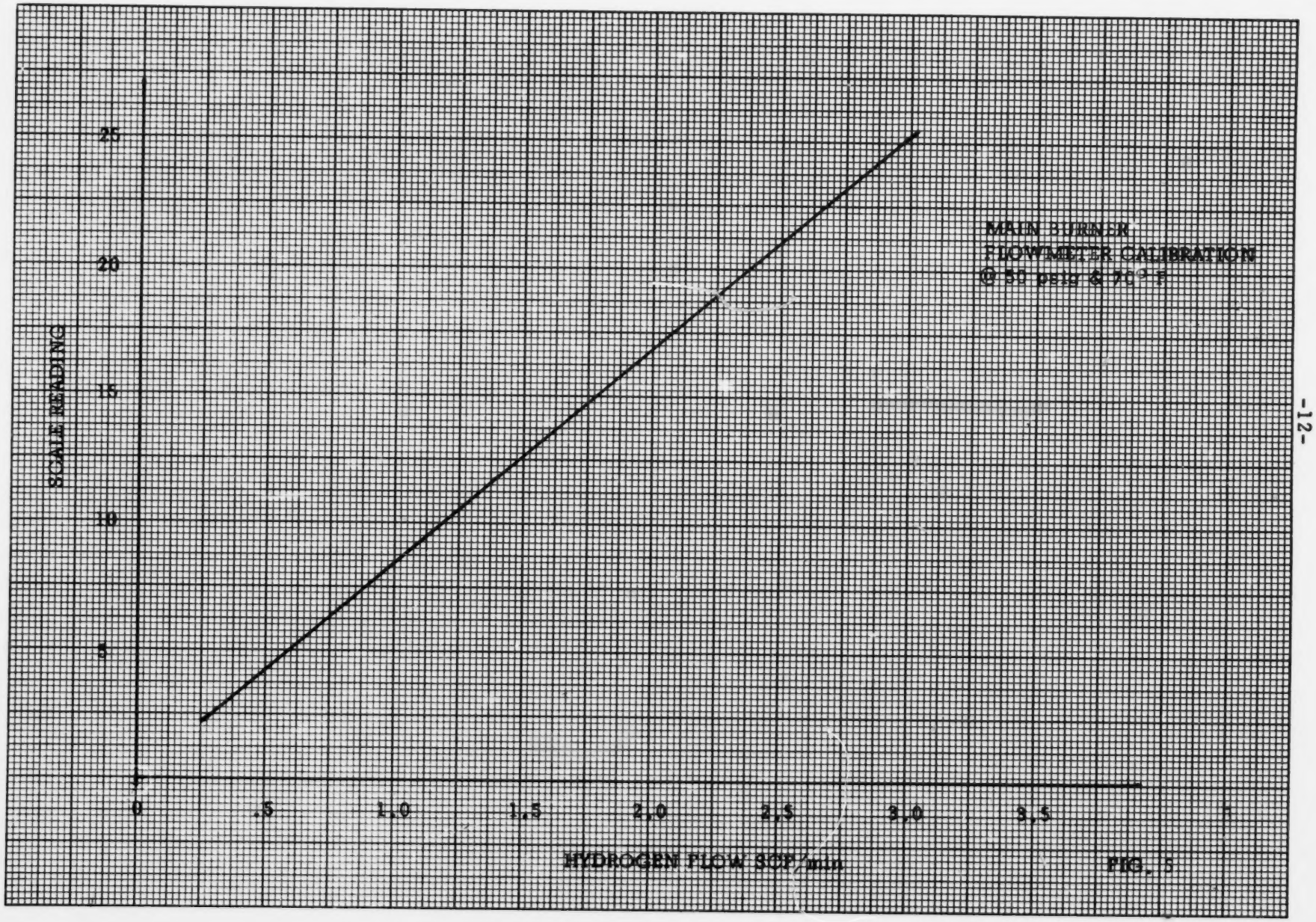


FIG. 5

K+E ... TO ... INCH ... 13:
7 X 10 INCHES MADE IN U.S.A.
KEUFFEL & ESSER CO.

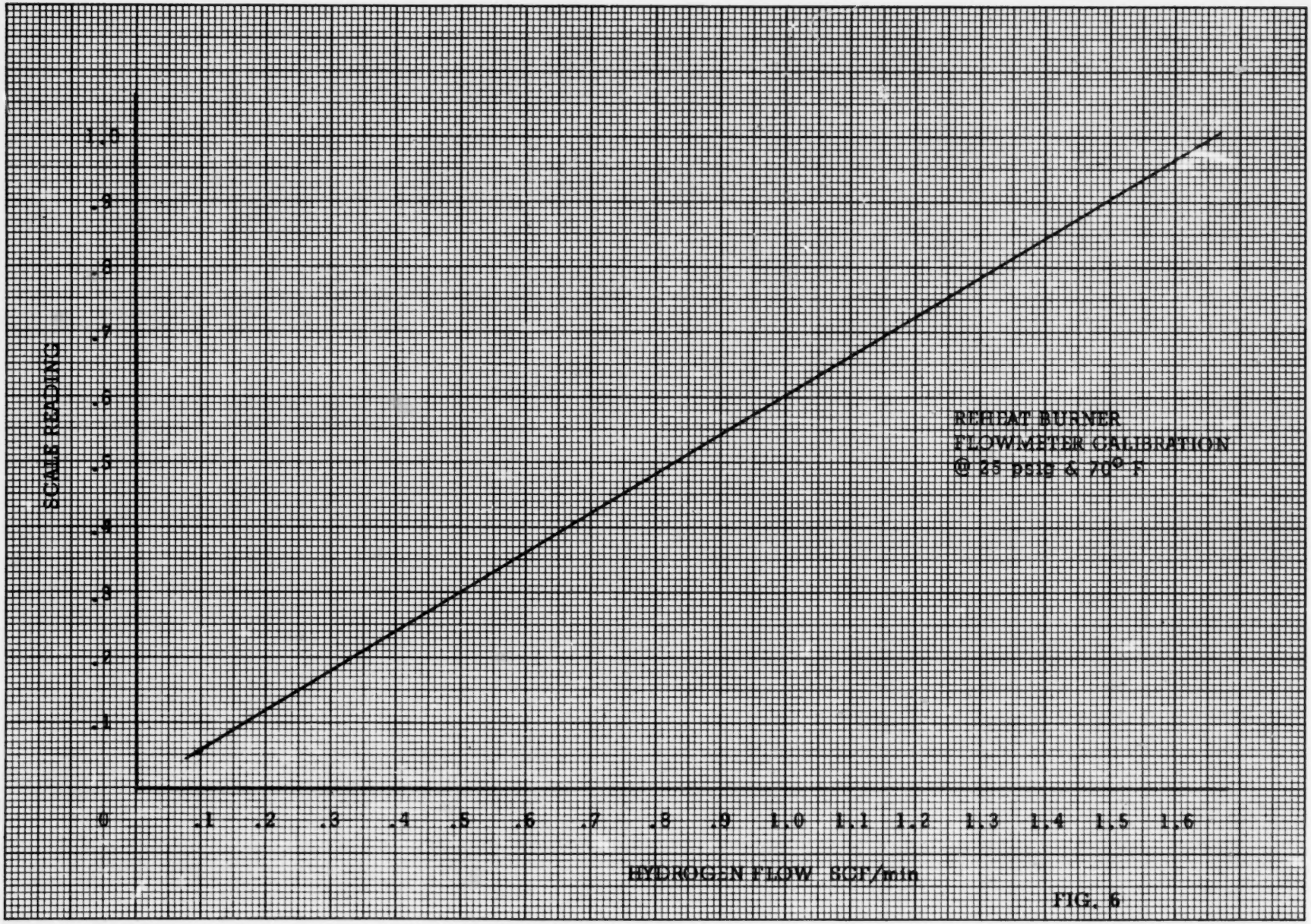


FIG. 6

KEUFFEL & ESSER CO.

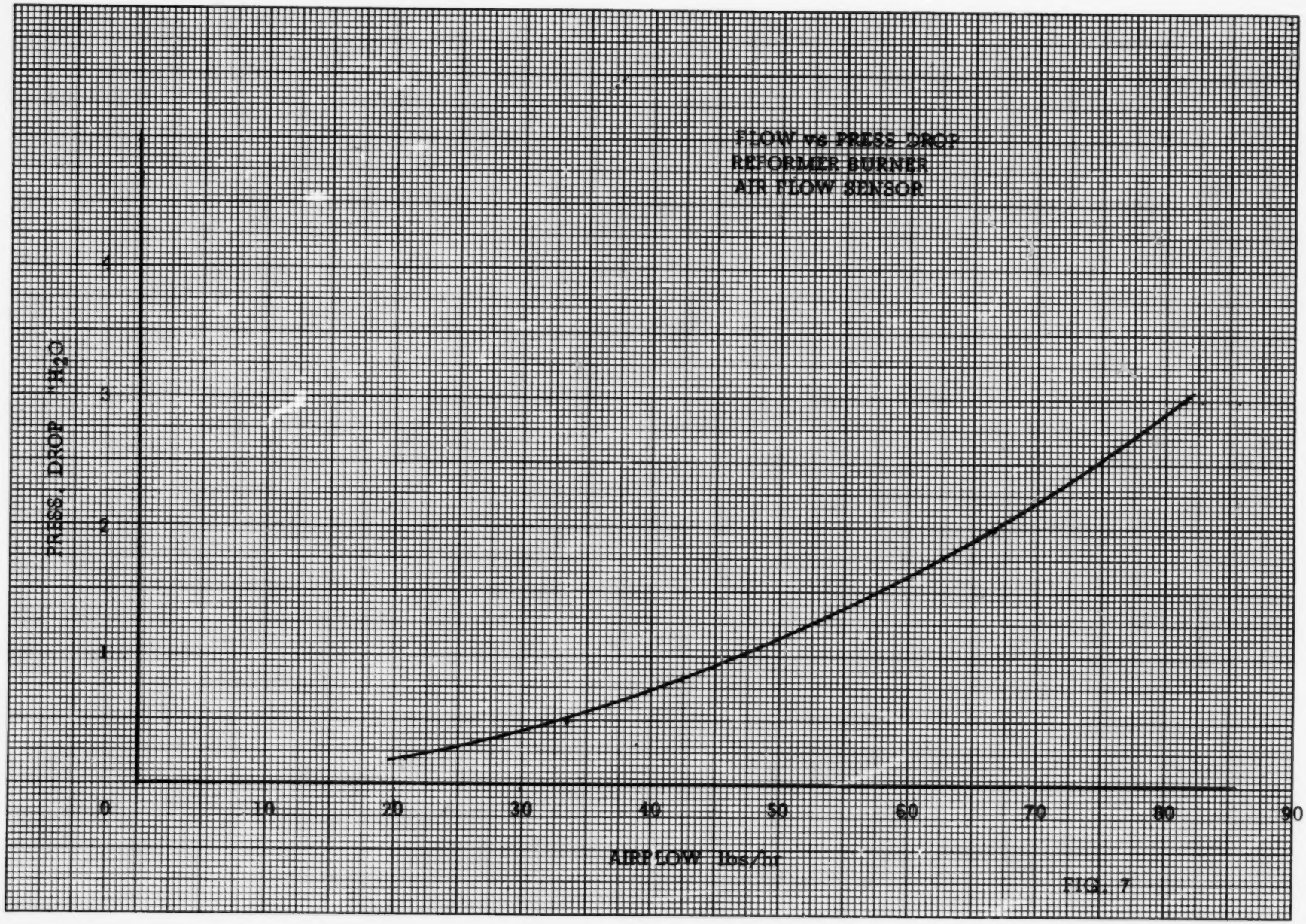


FIG. 7

condense the excess water and condensable hydrocarbons. The liquids were then collected in a reservoir and were periodically tapped off for visual inspection. The product gas was passed through a regulator valve which served to hold the process pressure at the selected value by modulating the flow orifice area. The flow rate was measured downstream of the regulator valve by passing the gas through a fixed orifice of known area and determining the gas temperature and pressure drop. From these data the flow was computed. Downstream of the regulator the gas passed through a flare tube to the atmosphere where it was burned. The flare tube was equipped with a "T" to which flasks were attached for drawing off gas samples for analysis.

5. Test Results

The graphic log of the endurance tests on No. 2 fuel oil and the product gas sample analysis are shown by Figures 8 and 9. The data sheets are shown by Figures 10, 11 and 12. The graphic log shows the reformer tube temperature, steam to mixing chamber temperature, the output gas temperature, the mixing chamber pressure, and the backpressure.

Inspection of the graphic log shows excursions in reformer tube temperature from 2070° F to 2160° F. It can be noted that there is a corresponding variation in the temperature of the steam to the mixing chamber. These temperature excursions are large enough to have an upsetting effect on the process and, because they occur together, indicate inadequate control of the reformer burner temperature. This is because the superheater, which supplies the steam to the mixing chamber, is heated by the same gas stream, supplied by the main burner, that heats the reformer tubes. The curve of temperature of the steam to the mixing chamber shows a dramatic drop after 5 1/2 hours of endurance operation. Subsequent to the test, the thermocouple was checked and inspected and was found to be malfunctioning. It is assumed that the failure occurred after 5 1/2 hours. The output gas curves show a drop of 100° F at the 4 1/4 hour point. There is no other data to corroborate this drop; and because of this, and because it is exactly 100° F below where it is expected to be, it is felt that it is a reading error.

The reformer pressure drop can be obtained by subtracting the back pressure from the mixing chamber pressure. Comparing the curves of these pressures it can be seen that this pressure drop is erratic and the variations increase toward the end of the test run. An increase in pressure drop across the reformer indicates partial blocking with deposited but unreformed carbon. An erratic pressure drop indicates a variation in the rates of carbon deposition and reformation. These variations in reformer pressure drop can

K&E 7 X 10 INCHES MADE IN U.S.A.
KEUFFEL & ESSER CO.

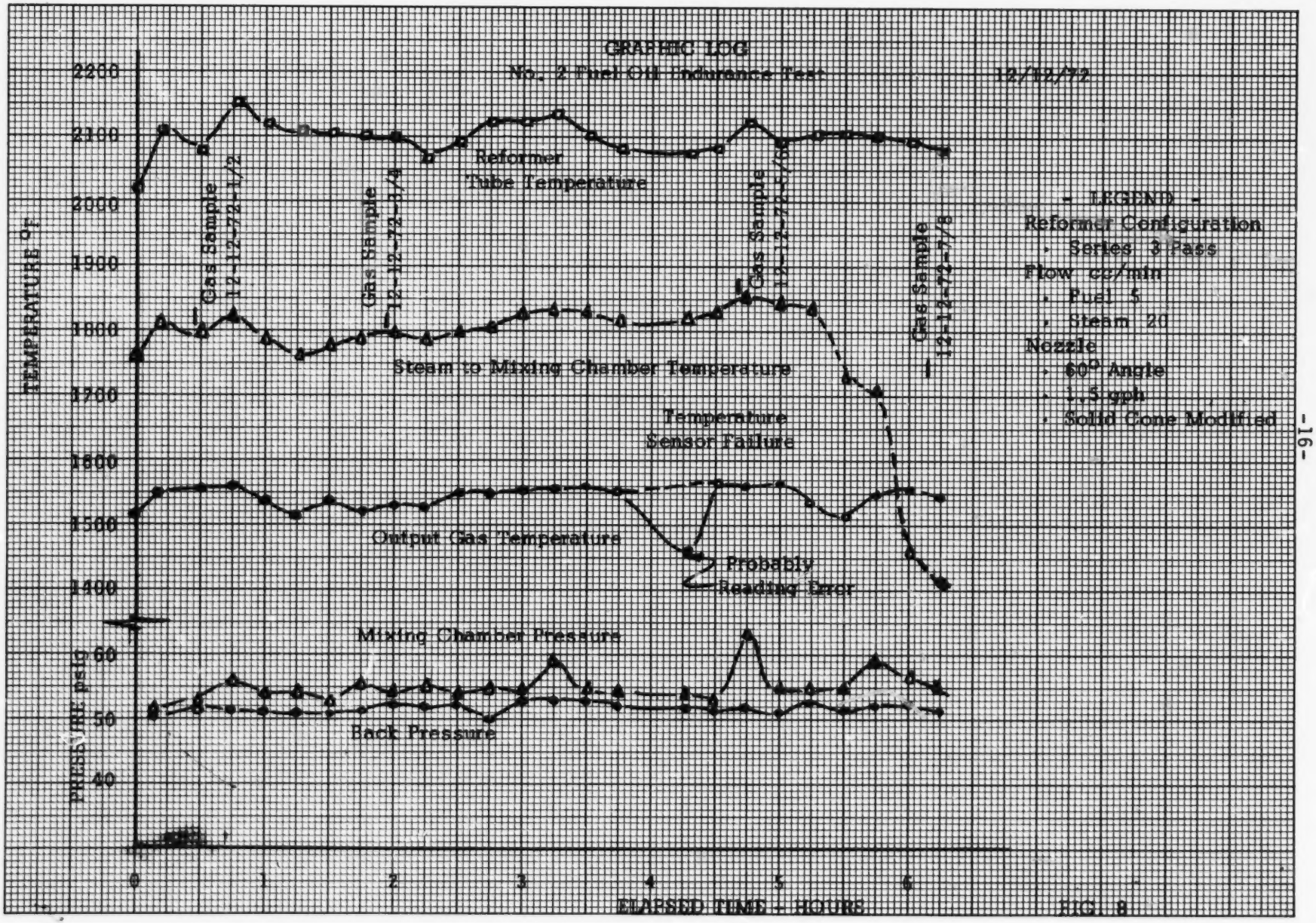


FIG. 8

PRODUCT
GAS SAMPLE ANALYSIS
CORRECTED TO BE FREE OF AIR & H_2O & NORMALIZED
No. 2 Fuel Oil $C_{7.3}$ $H_{12.16}$

Sample No.	H_2 %	CH_4 %	CO %	CO_2 %	C_2H_4 %	% Carbon Deficiency	H.H.V. Btu/cu ft
12-12-72-1	61.8	5.0	26.0	7.9	0	- 1.77	329.2
*12-12-72-2A	68.5	4.0	20.1	7.4	0.02	+36.6	322.7
*12-12-72-3	64.6	6.3	23.7	5.4	0.02	+30.9	344.0
12-12-72-4B	62.5	5.9	26.0	5.6	0.02	+15.7	340.7
*12-12-72-5C	67.2	4.7	20.7	7.4	0.02	+33.3	326.8
12-12-72-6C	64.6	4.5	22.5	8.1	0.34	+16.4	327.2
*12-12-72-7D	65.1	5.6	22.3	6.9	0.02	+27.7	334.6
12-12-72-8	62.9	5.4	24.8	6.8	0.01	+13.2	333.6

*Analysis by an outside testing laboratory

FIG. 9

Run No.:

REFORMER DATA SHEET

Date: 12-12-72

TEST OBJECTIVE: #2 FUEL OIL ENDURANCE

Sheet No. 1 OF 3

FUEL: # 2 FUEL OIL TIME ON: 9:36 TIME OFF:

NOZZLE: 60°-1.5 GPH-SOLID CONE HOO TUBE PACKING:

TIME		9:36	9:42	10:00	10:03	10:15	10:30	10:45	11:00	11:15
FLOWS:	Air Ref. # 1 ΔP IN MM OF H_2O ACROSS 7/8 IN. DIA ORIFICE	52	45	45	45	45	45	45	45	45
	Air Ref. # 2									
	Air Ref. # 3									
	H_2 Ref. # 1 SCALE READING FIG 5	1.5	2.0	2.0	2.0	2.0	2.0	2.0	2.0	2.0
	H_2 Ref. # 1 Reheat SCALE READING FIG 6	.30	.30	30	.30	30	.25	.25	.25	.30
	H_2 Ref. # 2									
	H_2 Ref. # 3									
	Main Water CC/MIN.	21	21	20	20	20	20	20	20	20
	Auxiliary Water									
	Oil CC/MIN.	7	7	5	5	5	5	5	5	5
TEMPS: °F	Ref. Tube #1	2030	2125	2080	2130	2160	2120	2110	2100	2100
	Ref. Tube #2									
	Ref. Tube # 3									
	Steam to Mix. Chamber	1770	1820	1800	1830	1835	1780	1765	1770	1800
	Output Ref. # 1	1520	1560	1560	1570	1565	1545	1515	1540	1530
	Auxiliary Steam									
	Output Reformer # 2									
	Output Reformer # 3									
Pressures: PSI	Oil Nozzle	-	56	54	59	58	54	56	54	59
	Mixing Chamber	12	52	53	57	57	53	55	53	57
	~~Output Ref. #1~~ H_2O TO SUPERHEATER	18	54	53	58	58	53	55	54	58
	Output Ref. #2									
	Input Ref. # 3			SAMPLE NO 12 12 72-14	SAMPLE NO 12 12 72-24					
	Cathedral Ref. # 3									
	Output Ref. # 3									
	Back Pressure	14	52	51	51	51	51	51	51	51

FIG 10

Run No.: REFORMER DATA SHEET Date: 12-12-'72

TEST OBJECTIVE: #2 FUEL OIL ENDURANCE Sheet No. 2 OF 3

FUEL: #2 FUEL OIL TIME ON: TIME OFF:

NOZZLE: 60°-1.5GPH-SOLID CONE-MOD. TUBE PACKING:

		TIME	11:30	11:45	12:00	12:15	12:30	12:45	13:00	13:15		
FLOWS:	Air Ref. # 1 ΔP IN MM OF H2O ACROSS A 1/16 DIA ORIFICE		45	45	45	45	45	45	45	45		
	Air Ref. # 2											
	Air Ref. # 3											
	H_2 Ref. # 1 SCALE READING FIG 5		1.8	1.8	1.8	2.0	2.0	2.0	2.0	1.8		
	H_2 Ref. # 1 Reheat SCALE READING FIG 6		.30	.30	.30	.31	.31	.33	.33	.33		
	H_2 Ref. # 2											
	H_2 Ref. # 3											
	Main Water CC/MIN.		20	20	20	20	20	20	20	20		
	Auxiliary Water											
	Oil CC/MIN		5	5	5	5	5	5	5	5		
TEMPS: °F	Ref. Tube #1		2100	2070	2095	2125	2120	2140	2100	2100		
	Ref. Tube #2											
	Ref. Tube # 3											
	Steam to Mix. Chamber		1800	1780	1800	1820	1840	1840	1830	1830		
	Output Ref. # 1		1535	1530	1550	1550	1560	1560	1560	1560		
	Auxiliary Steam											
	Output Reformer # 2											
	Output Reformer # 3											
Pressures: PSI	Oil Nozzle		54	58	54	57	54	64	54	57		
	Mixing Chamber		53	56	53	56	53	61	54	55		
	~~Output Ref. #1~~ H_2O TO SUPERHEATER		53	57	54	57	54	62	54	56		
	Output Ref. #2	SAMPLE N° 12-12-72-3M 4B										
	Input Ref. # 3											
	Cathedral Ref. # 3											
	Output Ref. # 3											
	Back Pressure		52	52	51	50	53	53	53	51		

FIG 11

Run No.:

REFORMER DATA SHEET

Date: 12-12-72

TEST OBJECTIVE: N°2 FUEL OIL ENDURANCE

Sheet No. 3 OF 3

FUEL: #2 FUEL OIL TIME ON: TIME OFF: 16:01

NOZZLE: 60°-1.5GPH · SOLID CONE, MOD. TUBE PACKING:

TIME		13:30	14:00	14:15	14:30	14:45	15:00	15:15	15:30	15:45
FLOWS:	Air Ref. # 1 ΔP IN MM OF H_2O ACROSS 7/8 IN DIA ORIFICE	45	45	45	45	45	45	45	45	45
	Air Ref. # 2									
	Air Ref. # 3									
	H_2 Ref. # 1 SCALE READING FIG 5	2.0	2.0	2.0	1.8	2.0	2.0	2.0	2.0	2.0
	H_2 Ref. # 1 Reheat SCALE READING FIG 6	.33	.33	.33	.33	.33	.16	.30	.30	.30
	H_2 Ref. # 2									
	H_2 Ref. # 3									
	Main Water CC/MIN.	20	20	20	20	20	20	20	20	20
	Auxiliary Water									
	Oil	5	5	5	5	5	5	5	5	5
TEMPS:	Ref. Tube #1	2075	2080	2130	2090	2105	2110	2110	2100	2085
	Ref. Tube #2									
	Ref. Tube # 3									
	Steam to Mix. Chamber	1830	1835	1860	1840	1845	1720	1700	1470	1420
	Output Ref. # 1	1470	1580	1560	1570	1540	1515	1550	1570	1545
	Auxiliary Steam									
	Output Reformer # 2									
	Output Reformer # 3									
Pressures:	Oil Nozzle	54	53	66	56	60	53	62	61	62
	Mixing Chamber	54	52	65	55	55	54	61	57	57
	~~Output Ref. #1~~ H_2O TO SUPERHEATER	54	52	66	56	57	52	62	58	59
	Output Ref. #2									
	Input Ref. # 3			SAMPLE N° 12 12 72-5C	SAMPLE N° 12 12 72-6C					SAMPLES N°s 12 12 72-7C/8C
	Cathedral Ref. # 3									
	Output Ref. # 3									
	Back Pressure	52	50	52	51	53	51	53	52	52

FIG 12

be seen to generally correlate with high reformer tube and steam to the mixing chamber temperatures, which are known to be critical to the rates of the decomposing and reforming reactions. Therefore, it is felt that better control of the burner temperature would have significantly improved the test results. The graphic log also includes notations of when gas samples were taken for chromatograph analysis. The unmarked analyses were performed on the gas chromatograph at International Materials and those indicated by an asterisk were performed by an independent outside laboratory. A copy of the laboratory report is attached, Fig. 13. This report shows the carbon monoxide concentration to be lower and the methane concentration to be higher than was shown by the analyses at International Materials. The descrepancy was found to be caused by these values being interchanged in the outside laboratory report. This is explained in their letter, Figure 14.

To evaluate the completeness of the process, a mass balance was performed using the chemical analysis of the fuel and the chromatograph analysis of the output reformed gas. The criterion developed to express the degree of completeness was percentage of carbon deficiency. This can be expressed:

$$\% \text{ Carbon Deficiency} = \frac{\text{Input Carbon} - \text{Output Carbon}}{\text{Input Carbon}} \times 100$$

An analysis showing zero carbon deficiency is for the reaction going to completion and an analysis showing some carbon deficiency indicates condensable hydrocarbons in the output product and therefore showing that the reaction has not proceeded to completion. The greater the carbon deficiency the less complete the reaction.

The graphic log for the No. 4 Fuel Oil Endurance Test is presented in Fig. 15. These curves show the reformer tube temperature, steam to mixing chamber temperature and output gas temperature to be maintained at close limits and a negligible variation in reformer pressure drop to exist throughout the test run. The test was interrupted after about 4 hours to replenish the gas supply system for the reformer burner. The shutdown period was approximately 45 minutes, during which the backpressure valve was disassembled and inspected. A light oily film was found on the parts, was wiped off and the unit was reassembled. The test proceeded to completion without incident. A tabulation of the output gas analysis from the No. 4 Fuel Oil Endurance Test is given on Fig. No. 16. As in the case with the tabulation of the No. 2 Fuel Oil Endurance Test, the samples indicated by asterisks were analyzed by an outside independent testing laboratory and the remainder were analyzed using a gas chromatograph at International Materials. These analyses also can be shown to be slightly

NEW ENGLAND ANALYTICAL & TESTING LABORATORY

2 SHADY OAK LANE
NATICK, MASS. 01760
(area 617) 873-8469

December 15, 1972

REPORT OF ANALYSIS

For: International Materials Corp.
Northwest Industrial Park
Second Avenue
Burlington, Mass. 01803

P.S.R. 28066

Att: Mr. Al. Able

Problem: To analyze four gas samples for the following components: H_2, O_2, N_2, CO, CH_4, CO_2, C_2H_4, and C_2H_6.

Instrument: Gas Chromatograph with Thermal Conductivity Detector.

Results:

Component	Sample Numbers			
	#2A	#3B	#5C	#7D
Hydrogen	69.523	62.524	64.857	66.724
Oxygen	0.036	0.036	0.140	0.039
Nitrogen	0.150	0.163	0.613	0.175
Carbon Monoxide	4.095	6.066	4.516	5.746
Methane	20.410	22.945	19.955	22.880
Carbon Dioxide	7.479	5.220	7.185	7.097
Ethylene	All samples had less than 0.02%			
Ethane	None observed in any sample.			

All samples were run in duplicate.

Respectfully submitted,

Lawrence C. O'Brien

Lawrence C. O'Brien

LCO'B;eo

FIG. 13

NEW ENGLAND ANALYTICAL & TESTING LABORATORY

2 SHADY OAK LANE
NATICK, MASS. 01760
(area 617) 873-8469

January 8, 1973

Mr. Jim Able
International Materials Corp.
Northwest Industrial Park
Second Avenue
Burlington, Mass. 01803

Dear Jim,

New England Analytical & Testing Laboratory has been analyzing Gas Reactor samples for International Materials since April of 1972, with satisfactory results.

Recently, N.E.A.T Lab. has analyzed two sets of four samples each, reported on December 15, 1972, and January 5, 1973. These results reported to International Material each contained an error as follows:

December 15, 1972 report. The figures calculated for the Methane, and Carbon Monoxide concentrations were interposed, i.e., the CO figures were reported for the CH_4, and vice versa. The figures should have read:

Component	SAMPLE NUMBERS			
	#2A	3B	5C	7D
Methane	4.095	6.066	4.516	5.746
Carbon Monoxide	20.410	22.945	19.955	22.880

These changes were verbally corrected with Chuck Paltz on December 17, 1972.

January 15, 1973 report. This report contained an abnormally high concentration of Carbon Dioxide. The source of the error was found to be a new instrument that was put into service on Jan. 3, 1973. The resistors of the attentuator in the #8, and #16 position were identical, instead of being 50% different. So, the figures reported were twice the concentration of what was actually present. The following figures are the real concentrations which should have been reported:

Component	Sample Numbers			
	#2	#3	#6	#7
Carbon Dioxide	6.512	6.710	5.830	7.975

Yours very truly,

Lawrence C. O'Brien

Lawrence C. O'Brien

LCO'B:eo

FIG. 14

K&E 10 X 10 TO ¼ INCH 7 X 10 INCHES 46 1325 MADE IN U.S.A.
KEUFFEL & ESSER CO.

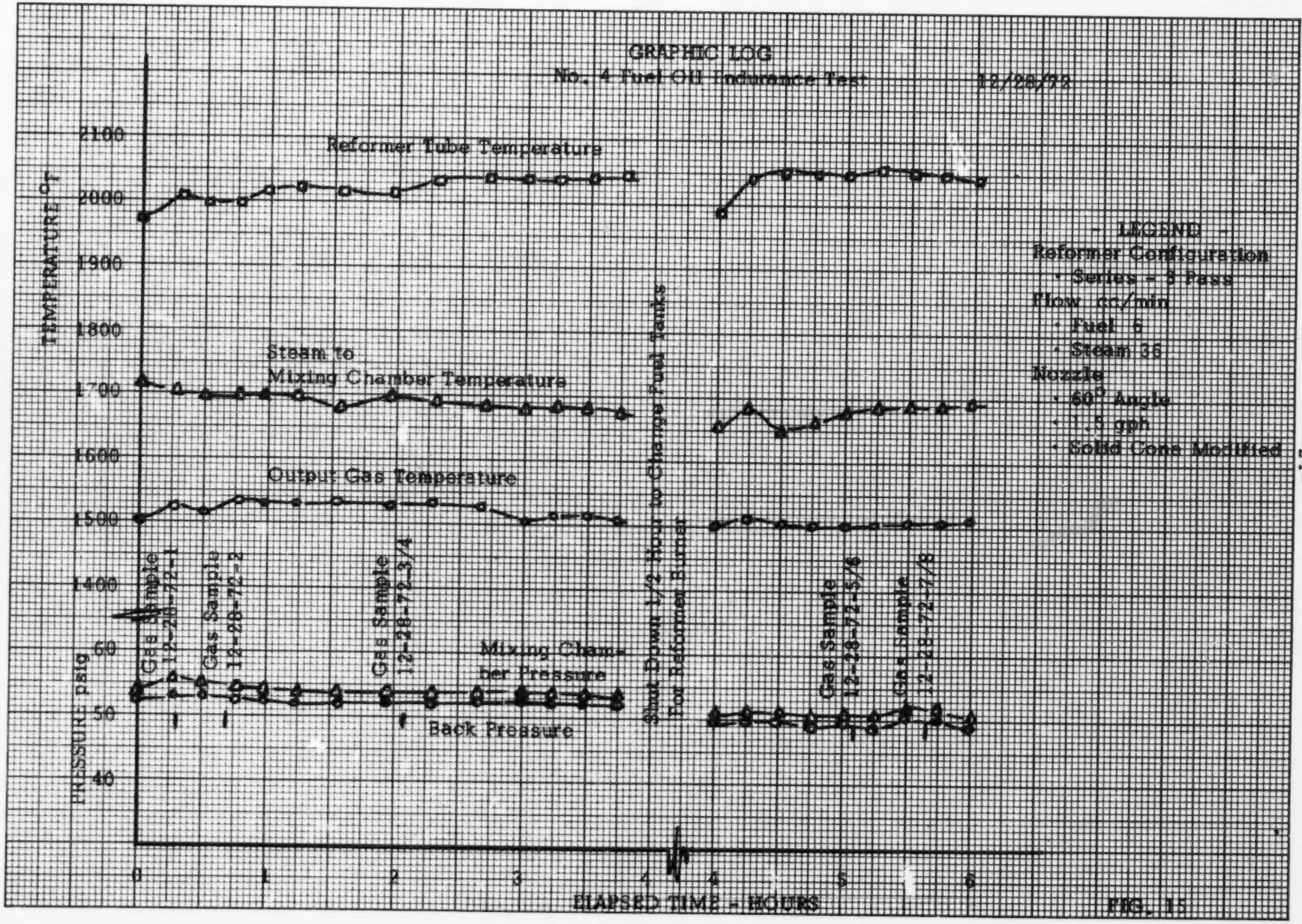


FIG. 15

PRODUCT
GAS SAMPLE ANALYSIS
CORRECTED TO BE FREE OF AIR & H_2O & NORMALIZED
No. 4 Fuel Oil $C_{7.25}$ $H_{12.23}$

Sample No.	H_2 %	CH_4 %	CO %	CO_2 %	C_2H_4 %	C_2H_6 %	% Carbon Deficiency	H.H.V. Btu/cu ft
12-28-72-1	62.4	6.1	23.6	7.9	0.02	0	+ 9.8	334.6
*12-28-72-2	61.6	7.1	24.1	7.2	0.08	0	+12.9	344.1
*12-28-72-3	61.7	7.0	23.6	7.6	0.07	0	+12.8	342.5
12-28-72-4	60.8	7.2	25.8	6.0	0.25	0	+12.2	351.1
12-28-72-5	60.3	7.1	27.3	5.0	0.25	0.04	+10.8	353.9
*12-28-72-6	61.9	7.0	24.2	6.8	0.08	0	+16.0	345.3
*12-28-72-7	60.0	8.0	23.4	8.6	0.034	0	+ 4.9	345.6
12-28-72-8	60.4	8.1	25.5	5.3	0.51	0.15	+18.4	364.9

*Analysis by an outside testing laboratory

FIG. 16

carbon deficient indicating that the reformer was not quite long enough to completely decompose and reform the No. 4 fuel oil. Copies of the test data sheets are included, Figs. No. 17, 18, 19 and 20. The report from the independent laboratory is shown, Fig. 21. This report shows the carbon monoxide concentration to be higher than was shown by the analyses at International Materials. The discrepancy was determined to have been caused by an incorrectly assembled attenuator in a new piece of test equipment being used for this work. This is explained in a letter from the vendor, Fig. 14. Laboratory analyses of the fuels used on this test and of the content of the ash are shown in Figs. 22 and 23.

The test program on No. 6 high sulfur residual oil was in the calibration phase when it was halted. Progress had been made toward establishing satisfactory endurance conditions but completely acceptable conditions had not yet been achieved.

Early in the calibration program it was found that No. 6 oil behaved differently in a reformer from No. 2 and No. 4 oil. There were both mechanical problems in that No. 6 oil was much harder to spray and process problems in that No. 6 oil had different kinetics on decomposing and reforming than No. 2 and No. 4 fuel oils.

The problem in spraying the fuel was in the availability of equipment to spray No. 6 high sulfur oil at the rates required by this program. There was an abundance of equipment available for spraying No. 6 oil in fine enough droplet sizes for the requirement of this program. However, the available equipment was designed for large power plant boiler applications and the minimum flow capability was much more than the maximum flow capability of the reformers used on this test. Some steam injection nozzles were investigated but their minimum steam flow requirement at the fuel flow that the reformer could handle resulted in an unacceptable steam-carbon ratio for the test.

Work was done at International Materials to find ways to use the process steam to break the fuel stream into acceptably small droplets to establish a reformer packing density that would control the rate of decomposing the oil within acceptable limits and to find ways to inject the superheated steam to achieve the desired high rate of reforming. However, at the time that the test was halted, the kinetics of processing No. 6 high sulfur residual oil were not well enough understood or controlled to permit a run of over about 1/2 hour before the reformer pressure drop became excessive. This required that the fuel be shut off and the reformer be cleaned out by purging with high temperature steam. There are many reformer operating conditions and combinations of conditions that remain to be explored and it is felt that a regime will be found where the decomposing and reforming

Run No.:

REFORMER DATA SHEET

Date: 12-28-72

TEST OBJECTIVE: #4 FUEL OIL ENDURANCE

Sheet No. 1 OF 4

FUEL: #4 FUEL OIL TIME ON: 12:06 TIME OFF:

NOZZLE: 60° CONE, SOLID - 1.5 GPH - MOD. TUBE PACKING:

TIME		12:06	12:25	12:30	12:36	12:41	12:54	1:04	1:19	
FLOWS:	Air Ref. # 1 ΔP IN INCHES OF H_2O ACROSS 1/8 IN. DIA ORIFICE	2.0	1.5	2.0	2.0	2.0	2.0	2.0	2.0	
	Air Ref. # 2									
	Air Ref. # 3									
	H_2 Ref. # 1 SCALE READING FIG 5	1.2	1.2	1.1	1.2	1.3	1.1	1.2	1.2	
	H_2 Ref. # 1 Reheat SCALE READING FIG 6	.45	.45	.45	.45	.45	.45	.45	.45	
	H_2 Ref. # 2									
	H_2 Ref. # 3									
	Main Water CC/MIN.	30	30	36	36	36	36	36	36	
	Auxiliary Water									
	Oil CC/MIN	0	0	6	6	6	6	6	6	
TEMPS:	Ref. Tube #1	1200	2030	1960	1980	2010	2020	2000	2000	
°F	Ref. Tube #2									
	Ref. Tube # 3									
	Steam to Mix. Chamber	800	1670	1720	1730	1750	1700	1700	1700	
	Output Ref. # 1	600	1420	1500	1510	1530	1530	1520	1540	
	Auxiliary Steam									
	Output Reformer # 2	12:15 RUGS WORKING		FLAME ON	CLEAN NOZZLE/ TEMPERATURE FLAME	SAMPLE NO 122872-1		SAMPLE NO 122872-2 WATER CLEAR		
	Output Reformer # 3									
Pressures:	Oil Nozzle	0	0	18	59	61	62	60	60	
PSI	Mixing Chamber	0	8	10	52	54	57	54	54	
	~~H_2O TO SUPERHEATER~~ Output Ref. #1	0	10	16	55	56	58	57	57	
	~~H_2O TANK PRESSURE~~ Output Ref. #2	100	100	100	100	100	100	100	100	
	~~FUEL BOOST PRESS.~~ Input Ref. #3	10	10	10	10	10	10	10	10	
	Cathedral Ref. # 3									
	Output Ref. # 3									
	Back Pressure	0	5	10	52	54	54	54	54	

FIG 17

Run No.:

REFORMER DATA SHEET

Date: 12-28-72

TEST OBJECTIVE: #4 FUEL OIL ENDURANCE

Sheet No. 2 OF 4

FUEL: #4 FUEL OIL TIME ON: TIME OFF:

NOZZLE: 60° CONE SOLID - 1.5GPH - MOD TUBE PACKING:

TIME		1:36	1:50	2:18	2:30	2:50	3:13	3:30	3:45	
FLOWS:	Air Ref. # 1 (ΔP IN INCHES OF H_2O ACROSS .38" DIA ORIFICE)	2.0	2.0	2.0	2.0	2.0	2.0	2.0	2.0	
	Air Ref. # 2									
	Air Ref. # 3									
	H_2 Ref. # 1 (SCALE READING FIG 5)	1.2	1.2	1.2	1.2	1.2	1.2	1.2	1.2	
	H_2 Ref. # 1 Reheat (SCALE READING FIG 6)	.45	.45	.45	.45	.45	.45	.45	.45	
	H_2 Ref. # 2									
	H_2 Ref. # 3									
	Main Water cc/MIN	36	36	36	36	36	36	36	36	
	Auxiliary Water									
	Oil cc/MIN	6	6	6	6	6	6	6	6	
TEMPS: °F	Ref. Tube #1	2025	2025	2025	2025	2040	2040	2040	2040	
	Ref. Tube #2									
	Ref. Tube # 3									
	Steam to Mix. Chamber	1700	1700	1680	1700	1700	1690	1690	1690	
	Output Ref. # 1	1540	1535	1540	1530	1540	1530	1500	1520	
	Auxiliary Steam									
	Output Reformer # 2			SMALL SPOT THE- TRACE OF THE AFTER WATER GEN. GOOD 12-28-72 3/4						
	Output Reformer # 3							WATER CLEAR		
Pressures: PSI	Oil Nozzle	60	60	60	60	60	60	60	60	
	Mixing Chamber	54	54	54	54	54	55	54	55	
	Output Ref. #1	57	57	57	57	57	58	57	57	
	Output Ref. #2	100	100	100	100	100	100	100	100	
	Input Ref. # 3	10	10	10	10	10	10	10	10	
	Cathedral Ref. # 3									
	Output Ref. # 3									
	Back Pressure	54	54	54	54	54	55	54	55	

FIG. 18

Run No.:

REFORMER DATA SHEET

Date: 12-28-72

TEST OBJECTIVE: #4 FUEL OIL ENDURANCE

Sheet No. 3 OF 4

FUEL: #4 FUEL OIL TIME ON: TIME OFF:

NOZZLE: 60° CONE, SOLID - 1.5 GPH - MOD. TUBE PACKING:

TIME		4:00	4:15	4:30	4:40	5:00	5:15	5:30	5:45		
FLOWS:	Air Ref. # 1 Δ PIN INCHES OF H_2O ACROSS 7/8" DIA. ORIFICE	2.0	2.0			2.0	1.8	1.8	2 3/8		
	Air Ref. # 2										
	Air Ref. # 3										
	H_2 Ref. # 1 SCALE READING FIG 5	1.2	1.2			1.8	1.4	1.4	1.4		
	H_2 Ref. # 1 Reheat SCALE READING FIG 6	.45	.45			.45	.45	.45	.45		
	H_2 Ref. # 2										
	H_2 Ref. # 3										
	Main Water CC/MIN.	36	36			36	36	36	36		
	Auxiliary Water										
	Oil CC/MIN.	6	6			6	6	6	6		
TEMPS:	Ref. Tube #1	2040	2050			1960	2050	2050	2050		
°F	Ref. Tube #2										
	Ref. Tube # 3										
	Steam to Mix. Chamber	1700	1690			1670	1700	1695	1685		
	Output Ref. # 1	1520	1510			1500	1510	1505	1500		
	Auxiliary Steam										
	Output Reformer # 2										
	Output Reformer # 3										
Pressures:	Oil Nozzle	60	60.5			57	57.5	58	58.5		
PSI	Mixing Chamber	55	55			50	51	52	49		
	~~Output Ref. #1~~ H_2O TO SUPERHEATER	58	58			53	54	55	56		
	~~Output Ref. #2~~ H_2O TANK PRESS.	100	100			100	100	100	100		
	~~Input Ref. #3~~ FUEL BOOST PRESS.	10	10			10	10	10	10		
	Cathedral Ref. # 3										
	Output Ref. # 3										
	Back Pressure	55	55			50	51	52	49		

SHUT DOWN TO CHANGE H_2 TANKS AND TO CLEAN BACKPRESSURE REGULATOR WHILE DOWN. FOUR HOURS CONTINUOUS RUNNING WITH NO PROBLEM

REFORMER BACK ON - FUEL OFF
FUEL ON AT 5:00 PM

SAMPLE(S) TAKEN 12:28:72

FIG. 19

Run No.:

REFORMER DATA SHEET

Date: 12-28-72

TEST OBJECTIVE: #4 FUEL OIL ENDURANCE

Sheet No. 4 OF 4

FUEL: #4 FUEL OIL TIME ON: TIME OFF: 7:01

NOZZLE: 60° CONE, SOLID - 1.5 GPH - M00. TUBE PACKING:

TIME		6:00	6:15	6:30	6:45	7:00					
FLOWS:	Air Ref. # 1 ΔP IN INCHES OF H_2O ACROSS A 7/16" DIA. ORIFICE	2.0	2.0	2.0	2.0	2.0					
	Air Ref. # 2										
	Air Ref. # 3										
	H_2 Ref. # 1 SCALE READING FIG 5	1.4	1.4	1.4	1.4	1.4					
	H_2 Ref. # 1 Reheat SCALE READING FIG 6	.485	.485	.485	.46	.46					
	H_2 Ref. # 2										
	H_2 Ref. # 3										
	Main Water CC/MIN	36	36	36	36	36					
	Auxiliary Water										
	Oil CC/MIN	6	6	6	6	6					
TEMPS:	Ref. Tube #1	2050	2055	2050	2050	2040					
°F	Ref. Tube #2										
	Ref. Tube # 3										
	Steam to Mix. Chamber	1680	1690	1695	1690	1700					
	Output Ref. # 1	1500	1510	1505	1510	1570					
	Auxiliary Steam										
	Output Reformer # 2				SAMPLES/OS 12-28-72 4/8						
	Output Reformer # 3										
Pressures:	Oil Nozzle										
PSI	Mixing Chamber	52	50	54	52	50					
	~~H_2O TO SUPERHEATER Output Ref. 1~~	56	54	56	56	56					
	~~H_2O TANK PRESSURE Output Ref. 2~~	100	100	100	100	100					
	~~FUEL PUMP PRESS. Input Ref. 3~~	10	10	10	10	10					
	Cathedral Ref. # 3										
	Output Ref. # 3										
	Back Pressure	52	50	54	52	50					

FIG. 20

NEW ENGLAND ANALYTICAL & TESTING LABORATORY

2 SHADY OAK LANE
NATICK, MASS. 01760
(area 617) 873-8469

January 5, 1972

REPORT OF ANALYSIS

For: International Materials Corp.
Northwest Industrial Park
Second Avenue
Burlington, Mass. 01803

P.O.# Mr. Sorensen's Letter

Att: Mr. Harold P. Sorensen
Engineering Mgr.

Problem: To analyze four gas samples labeled #122872-2, 3, 6 & 7, for the following components: Hydrogen, Oxygen, Nitrogen, Carbon Monoxide, Methane, Carbon Dioxide, Ethylene, Ethane, and Carbonyl Sulfide, and Hydrogen Sulfide.

Instrument: Gas Chromatograph with Thermal Conductivity Detector, and Linear Temperature Programmer.

Results:

	Sample numbers			
Component	#2	#3	#6	#7
Hydrogen	55.365	54.752	53.322	55.774
Oxygen	0.029	0.202	0.281	0.145
Nitrogen	0.129	0.841	1.273	0.587
Methane	6.347	6.206	6.037	7.419
Car.Monoxide	21.638	20.976	20.866	21.749
Car.Dioxide	13.024	13.421	11.660	15.950
Ethylene	0.073	0.060	0.070	0.032
Ethane	None Resolved			
Hy.Sulfide	None detected by response or odor.			
Car.Sulfide**	2-4ppm.	2-4ppm.	4-6ppm.	8-10ppm.

** Identity estimated by retention time plots, and odor.(No standards with samples.)

Remarks: All samples run in duplicate. Results called into Mr. Sorensen the morning of January 2, 1973.

Respectfully submitted,

Lawrence C. O'Brien

LCO'B;eo

FIG. 21

FUEL SAMPLE ANALYSIS

Conducted by

WALBAR LABORATORIES

Sample	No. 2 Fuel Oil	No. 4 Fuel Oil	No. 6 Residual Oil
C	87.58	86.94	85.83
H_2	12.16	12.23	11.29
S	00.00	00.40	2.05
N	00.054	00.126	0.416
BTU/lb.	19,464	19,317	18,570
Specific Gravity	.85571	.89190	.95805
Viscosity	33.9° API	27.2° API	16.2° API

FIG. 22

...ylcon, Inc.
Wide spectrum analytical & consulting services
P.O. Box 86
Boston, Mass. 02122
Tel. 617 287-0200

January 23, 1973

CLIENT: International Materials Co.
Northwest Industrial Park
Second Avenue
Burlington, Massachusetts 01803

Attention: Mr. Sorensen

CASE NO. 1417

PURPOSE OF TEST: Chemical analysis of two (2) samples of fuel oil

SAMPLE IDENTIFICATION: Sample marked: #4, #6

METHOD OF TEST: Ash Content: A.S.T.M D482
Ash Composition: Emission Spectroscopy

RESULTS:

	#4	#6
Ash % By Weight	0.07	0.09
Composition Of Ash:		
Silicon	D	3E
Vanadium	3E	D
Nickel	E	3E
Tin	3F	F
Iron	F	F
Sodium	F	E
Lead	F	F
Zinc	F	F
Aluminum	3G	3F
Calcium	3G	3F
Copper	3G]	3G
Chromium	3G	3G
Magnesium	3G	F
Barium	G	G
Titanium	G	3G

FIG. 23

January 23, 1973

International Materials Co.
Case No. 1417

Composition Of Ash: (Continued)	#4	#6
Silver	3G	3G
Manganese	3H	3H
Molybdenum	--	G
Strontium	--	3H

KEY: A = Greater Than 10%
B = 1 - 10%
C = .1 - 1.0%
D = .01 - .1%
E = .001 - .01%
F = .0001 - .001%
G = Less Than .0001%
3B = Three Times Letter Value

COMMENT: H < .00001%

Silica and vanadium were the major ingredients in the #4 oil. Vanadium, nickel and silicon were the major ingredients in the #6 oil.

Respectfully submitted,

ANYLCON, INC.

Robert A. Sullivan/pr
Robert A. Sullivan
Presdient

RAS:pr

kinetics will be compatible and stable reformer operation will be possible with the reaction going to completion.

6. Variations in the Test Results

The differences in the normalized air and water free gas sample analyses tabulated in Figures 9 and 16 can be attributed to actual differences in the gas samples and to variations in the gas analysis and data reduction techniques. The differences in the gas samples are due primarily to the difference in temperature at which the process was being run at the time that the samples were taken.

The graphic log for the No. 2 fuel oil test, Figure No. 8, shows significant differences between the steam temperatures and between the reformer tube temperatures when the various gas samples were taken. Because reactions taking place in the process are strongly temperature dependent, the product gas composition is also strongly temperature dependent. Thus, the differences in the process temperature at the time that the samples were taken can be expected to produce a significant difference in composition of the gas samples. The process temperature control was closer on the No. 4 fuel oil run than it was on the No. 2 fuel oil run as can be seen by comparing the graphic logs for the two tests, Figures Nos. 8 and 15. Comparing the gas analysis tabulations for these tests, Figures 9 and 16, shows a much smaller difference in gas composition on the No. 4 fuel oil run than on the No. 2 fuel oil run. The smaller difference is attributed to better process temperature control.

In addition to the difference in gas composition, there are differences in the gas analysis due to differences in operating the gas chromatograph and in interpreting the results. Gas chromatography compares the type and concentration of constituents in a sample gas to the concentration of the same constituents in a calibration gas. The output is a series of peaks on a strip chart. To minimize the chance of error due to nonlinearity in the equipment, the calibration gas sample is made up to be as near to the expected concentration as is possible. Because the calibration gas sample is the reference for the analysis, it is necessary that its constituents be accurately known. The calibration gas is made by injecting into a container each constituent at a fixed temperature and at a pressure that is a function of its desired partial pressure in the mixture. Thus, the limitations of pressure instrumentation dictates that the percentage error in each constituent in the calibration gas is an inverse function of the proportion of that constituent in the whole. Therefore, a larger error can be expected in the minor constituents than in the major constituents in the calibration. This error, which will appear in the analysis of unknown samples, can be expected

to vary linearly from as much as ± 2.5% for constituents that are 10% of the whole to ± 0.4% for constituents that are 60% of the whole.

Another variation in gas chromatograph analysis is in the method by which the gas samples are injected into the chromatograph. The chromatograph at International Materials has an automatic gas sampling valve through which samples of a fixed volume are injected. Because the process is automatic, there is no change for error due to differences in operator technique. The temperature of the gas samples being injected is held to ± 1° F and, because the reference is absolute zero, this will be responsible for an error of less than ± .25% in the output. The samples are injected into the chromatograph at atmospheric pressure. Because calibration samples are run daily, the error due to pressure differences will be that caused by the daily variation in barometric pressure, which seldom is as much as one inch of mercury. Because the reference is absolute pressure, the error from this source will generally be less than one part in 30 or about ± 3%. The temperature and pressure effects will act on all of the constituents proportionally. Thus, a change in atmospheric pressure or sample temperature between calibrating the chromatograph and running the test will cause the total of the test analysis to deviate from 100%, but each of the constituents will be the correct proportion of the whole. Therefore, normalizing the result will eliminate the variations due to changes in atmospheric pressure and sample temperature.

The third source of error is that associated with reducing the data from the strip chart. The output of the chromatograph appears as a series of peaks on a strip chart recording. The position of the peak with respect to the time at which the sample was injected into the chromatograph reveals the constituent and the area under the peak determines the amount of the constituent. In practice, to avoid tedious procedures for determining the areas under the peaks, the amount of the constituent is approximated by using the height of the peak as an indication of the area under the peak. This "peak height determination" method is a recognized and widely used technique in gas chromatography and has been shown to give a one sigma error distribution of ± 4%.

As an overall check on the procedure at International Materials, a gas sample was carefully made up by a competent external supplier and was treated as an unknown. The result of this test is shown in the following table:

Constituent	International Materials Analysis	Actual
H_2	55.379	54.9
O_2	.997	1.0
N_2	3.918	3.8
CH_4	10.004	9.8
CO	19.258	20.1
CO_2	6.082	6.2
C_2H_4	2.015	2.1
C_2H_6	2.189	2.2

The procedure used by the independent outside laboratory was similar to that used at International Materials except that the manual syringe method of injecting gas samples into the chromatograph was employed and their chromatograph was not calibrated on a daily basis.

The syringe method of injecting gas samples is a recognized technique and can be done with a high degree of accuracy by a skilled operator. The operators at the independent outside laboratory have the skill required for accurate work using syringe injection. Thus, it is felt that this procedure did not have a significant effect on the accuracy of their results. In spite of the fact that the outside laboratory did not calibrate their equipment daily, they did analyze a test sample gas every day to verify that their equipment was functioning properly. This is felt to be a reasonable check. Thus, their lack of daily calibration is not expected to have a significant effect on their results.

The following conversion table and plot, Figures 24 and 25, will be helpful in converting from the English units used on this program to the metric system.

CONVERSIONS

English		Metric
Cubic Feet	X 28.32 =	Liters
Pounds	X 453.6 =	Grams
Pounds per square inch	X 7.031×10^2 =	Kilograms per square meter
Inches	X 2.540 =	Centimeters

Fig. 24

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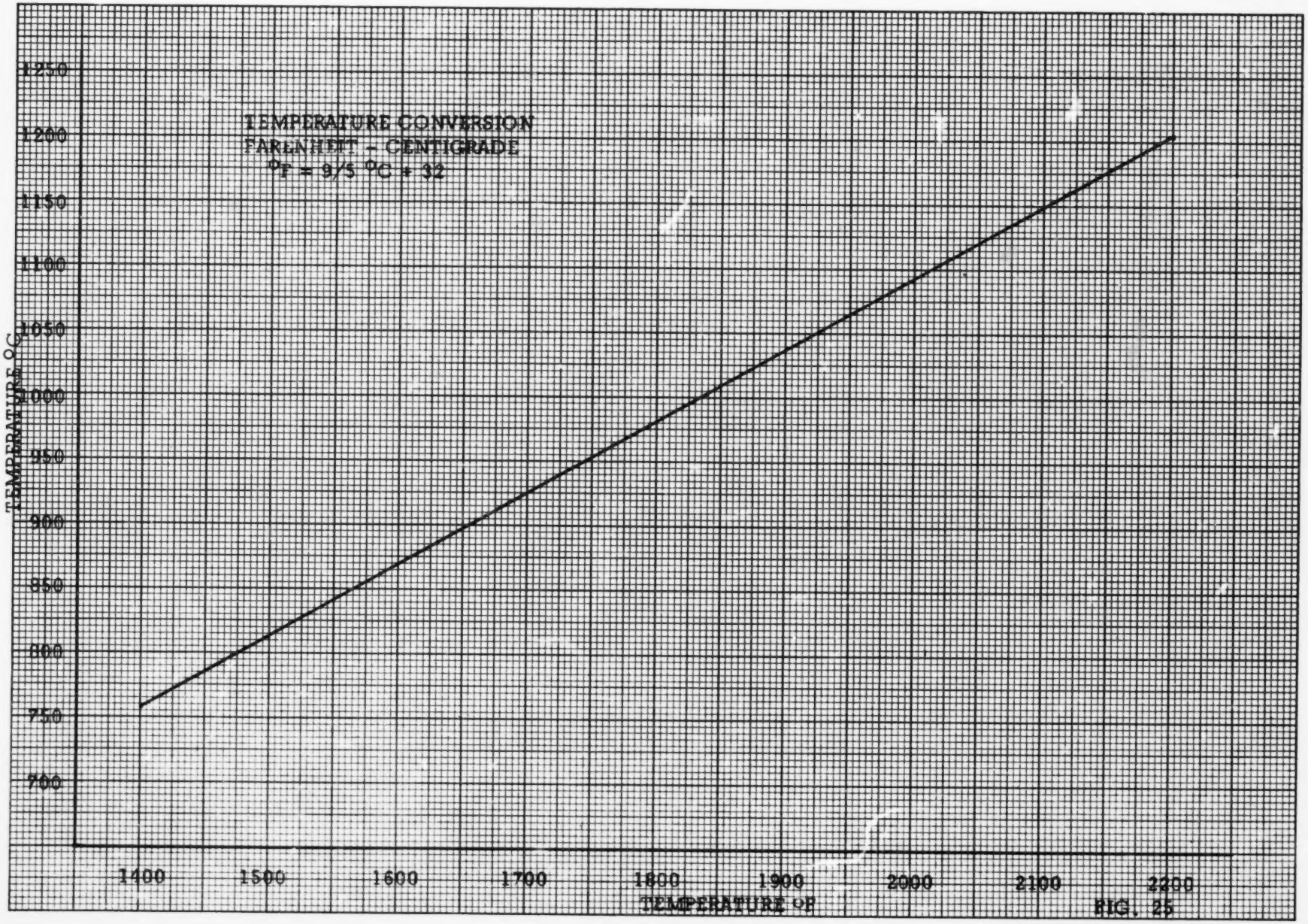

FIG. 25